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## MODIFICATION OF THE QUALITATIVE PROCEDURE FOR LEAD<sup>1</sup>

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The classes in general chemistry at Monticello A. and M. College are given, toward the end of the year, a brief course in elementary qualitative analysis for the purpose of impressing the student more strongly with some of the characteristics of the more common metals about which he has studied, and also to aid him in acquiring a more polished laboratory technique. In the scheme of analysis which he follows, however, we have, for the purpose of simplification, omitted from group two the entire tin sub-group; therefore, the metals arsenic, antimony, and tin are placed in no unknown that he will run.

Because of the lack of sufficient ventilation in the general chemistry laboratory, we found it highly desirable to devise a method by which we could eliminate that part of the procedure in the second group in which the solution is boiled down to fumes with concentrated sulphuric acid in order to separate lead from bismuth, copper, and cadmium. That could be done if lead were precipitated completely in the first group, thus removing the necessity of dealing with it in the second.

As lead precipitates more completely in a colder solution, experiments on the precipitation of lead from a solution cooled to a temperature of about 4°C. by means of the ice bath were carried out by adding hydrochloric acid to the chilled solution dropwise and with stirring, and a much more complete precipitation was obtained than was possible under ordinary conditions. The precipitation was not yet sufficiently complete to prevent some lead sulphide from being precipitated in the second group, but that was found to give no trouble.

Since both lead and bismuth, when present in the second group, are precipitated by ammonia and since small quantities of lead do not interfere with the sodium stannite test for bismuth, if lead is found in the first group the evaporation with sulphuric acid in the second group may be omitted without further alteration of procedure. However, although the precipitation of lead in chilled solution by means of hydrochloric acid is sufficiently complete for most purposes, yet if lead were present in only very small quantities or if ammonium or acetate ions were in the original solution, the lead might escape detection. Therefore it is desirable to have a method of separating lead from bismuth in the second group.

It was found possible to separate the precipitated lead and bismuth by treating the hydroxides on the filter paper with 5 N acetic acid. Such treatment of bismuth hydroxide alone did not dissolve enough bismuth to be precipitated by hydrogen sulphide, nor did this treatment prevent the precipitate from giving positive results with the sodium stannite test. Also, since potassium dichromate gives no precipitate with bismuth in acid solutions, whatever bismuth might come through with acetic acid would not interfere with the lead test.

To test the new procedure a solution containing lead, bismuth, copper, and cadmium was prepared. This solution was chilled and hydrochloric acid was added to remove the lead. After filtration, the solution was made alkaline with ammonia and was carried through the revised procedure from that point on. This procedure gave entirely satisfactory results at every step.

<sup>1</sup> Presented at the 1939 meeting.

The following scheme of analysis for group one and the copper sub-group of group two embodies the changes in procedure for lead.

Take 10 cc. of the original solution, chill in an ice bath, and add 5 N HCl dropwise with stirring until precipitation is complete. Filter and test the precipitate in the regular manner for the first group metals.

Neutralize the filtrate and add 5 cc. 3 N HCl, dilute to 50 cc. so that 0.3 N HCl solution is obtained. Treat with  $H_2S$ . Then transfer the copper group residue to a beaker containing 15 cc. of 5 N  $HNO_3$ , and an equal volume of water. Boil for two minutes, filter, and test the residue for mercury.

Make the filtrate alkaline with 5 N  $NH_3$  and filter. If the filtrate turns deep blue, the presence of copper is indicated and the blue color must be destroyed with potassium cyanide. Test for cadmium with  $H_2S$ .

Wash the precipitate from the ammonia treatment with water and discard the washings. If lead has been found in the first group test the residue with sodium stannite, a black coloration confirming the presence of bismuth. If lead has not been found and traces must be sought, or if ammonium and acetate ions are not known to be absent, wash with 5 cc. 5 N acetic acid repeatedly, add 5 cc. of water to the washings and test with potassium dichromate. Then test the residue for bismuth with sodium stannite solution.

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